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Thermal Exposure and Environment Effects on Tension, Fracture and Fatigue of 5XXX Alloys Tested in Different Orientations

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6. AUTHOR(S)
John J Lewandowski

5d. PROJECT NUMBER

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13. SUPPLEMENTARY NOTES

14. ABSTRACT
The work involved a systematic study to determine the effects of changes in temper (e.g. H131, H116, H128), thermal exposure conditions (i.e. time, temperature), and environment (e.g. dry air, humid air, solutions) on the environmental cracking susceptibility at different loading rates in both the S-T and L-T orientations. Experiments were conducted using slow strain rate tension (SSRT), fatigue crack growth using dcPD, and experiments conducted on fatigue precracked samples to determine the effects of changes in loading rate on cracking susceptibility. In addition, access to high resolution tomography occurred via a visit to the Harwell Diamond Light Source (DLS), UK, where in-situ cracking experiments were conducted as well as tomography experiments on previously tested samples. Regimes of EAC susceptibility were determined and the effects of changes in loading rate and solution on this susceptibility were determined.

15. SUBJECT TERMS
Al-Mg alloys, Sensitization, Environmentally Assisted Cracking (EAC), Slow Strain Rate Testing (SSRT), Fatigue Crack Growth (FCG)

16. SECURITY CLASSIFICATION OF: a. REPORT	b. ABSTRACT	c. THIS PAGE	17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON	19b. TELEPHONE NUMBER *(Include area code)*
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Contract Information

Contract Number	N00014-14-1-0593
Title of Research	Thermal Exposure and Environment Effects on Tension, Fracture and Fatigue of 5XXX Alloys Tested in Different Orientations
Principal Investigator	John J Lewandowski
Organization	Case Western Reserve University

Technical Section

Technical Objectives

The objectives of this work were to determine the effects of thermal exposure temperature and time on the environmentally induced cracking behavior of 5XXX monolithic aluminum alloys developed for naval applications. This type of approach, equipment and testing capabilities available at CWRU can address similar issues in other metallurgically relevant materials (e.g. Mg, Ti, etc.) if interest evolves in those systems.

5XXX Aluminum alloys are generally considered non-heat treatable alloys, as they contain solid solution additions of Mg. 5XXX alloys generally possess good corrosion characteristics, ductility, and toughness, depending on the temper designation. The annealed temper (i.e. O temper) is the lowest strength condition, and could represent the temper likely present near the heat affected zone of a welded 5XXX structure, or near the nodes in a truss structure. The H321 temper applies to products that are strain hardened after processing, while the H116 temper applies to products that acquire some strain hardening during working at elevated temperatures. Each of these tempers could be relevant in a naval structure, depending on the fabrication and joining technique utilized.

Other issues of relevance to 5XXX alloys are aspects related to sensitization of this material at low temperatures (e.g.$\leq$ 100 ^{o}C) for long periods of time (e.g. up to 2+ years). These types of exposures may change the mechanical behavior, including their susceptibility to environmentally assisted cracking (EAC). Very little of this information exists for thermal exposures and times relevant to actual applications in the literature, although such exposures will be relevant for any naval structures manufactured from 5XXX-type alloys.

Our work involved a systematic study to determine the effects of changes in temper (e.g. H131, H116), thermal exposure conditions (i.e. time, temperature), and environment (e.g. dry air, humid air, solutions) on the environmental cracking susceptibility at different loading rates in both the S-T and L-T orientations. Experiments were conducted using slow strain rate tension (SSRT), fatigue crack growth using dcPD, and experiments conducted on fatigue precracked samples to determine the effects of changes in loading rate on cracking susceptibility. In addition,

access to high-resolution tomography occurred via a visit to the Harwell Diamond Light Source (DLS), UK, where in-situ cracking experiments were conducted as well as tomography experiments on previously tested samples. Regimes of EAC susceptibility were determined and the effects of changes in loading rate and solution on this susceptibility were determined.

Technical Approach

The technical approach followed in this completed work has been to conduct quasi-static fracture and fatigue experiments on 5XXX commercial aluminum alloys of interest to the Navy, shown in Figure 1.

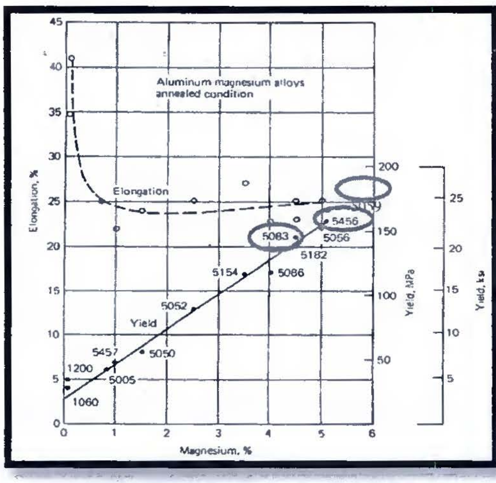


Figure 1: Typical 5XXX alloys and those tested in this work (i.e. 5456, 5380, 5059).

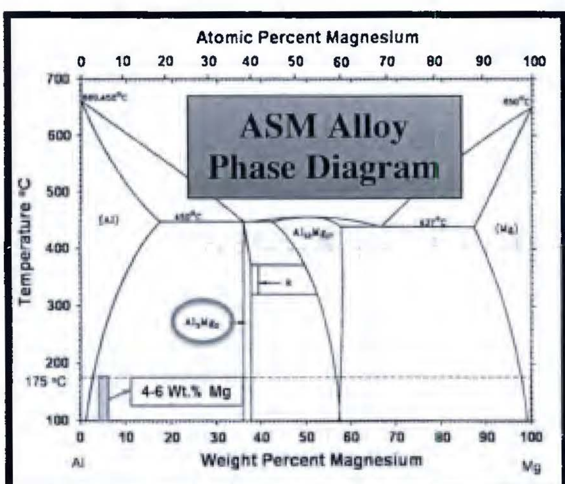


Figure 2: Al-Mg phase diagram showing typical Mg range for 5456, 5083, and 5059 alloys as well as Mg-rich equilibrium phase that can evolve at grain boundaries during sensitization.

Although 5083/5456/5XXX alloys are not precipitation hardened, the exposure of these materials at intermediate test temperatures for long periods of time results in the precipitation of a Mg-rich phase at the grain boundaries, Figure 2. This has been shown to significantly affect the mechanical behavior. We have conducted experiments where the 5083/5456/5XXX aluminum alloys are 'sensitized' by thermally exposing them to a range of intermediate temperatures (e.g. 60°C - 175°C) for very long periods of time (e.g. up to 4+ years). In addition to characterizing the microstructures with SEM and TEM, the effects of changes in strain rate and loading rate on the SSRT and EAC behavior in different solutions has been determined in both the S-T and L-T orientations. Comparisons have been made to the non-sensitized materials, and any changes in behavior have been documented with SEM fractography at the appropriate size scales and magnification. In addition, high-resolution tomography experiments were conducted at the DLS in order to image sub-surface damage in failed samples as well as conduct in-situ EAC experiments on sensitized material.

Final Progress Statement Summary

A variety of 5XXX series aluminum alloys (e.g. 5456, 5083, 5059) have been tested in a variety of temper conditions (e.g. H131, H116) in addition to examining the effects of sensitization on the quasi-static and EAC resistance using fatigue crack growth, SSRT, and fatigue precracked experiments in a variety of environments (e.g. dry air, humid air, solutions). High resolution SEM was utilized on materials given laboratory sensitization treatments for times up to 40,000 hours. Figure 3 shows 3D Optical and EBSD imaging of as-received materials.

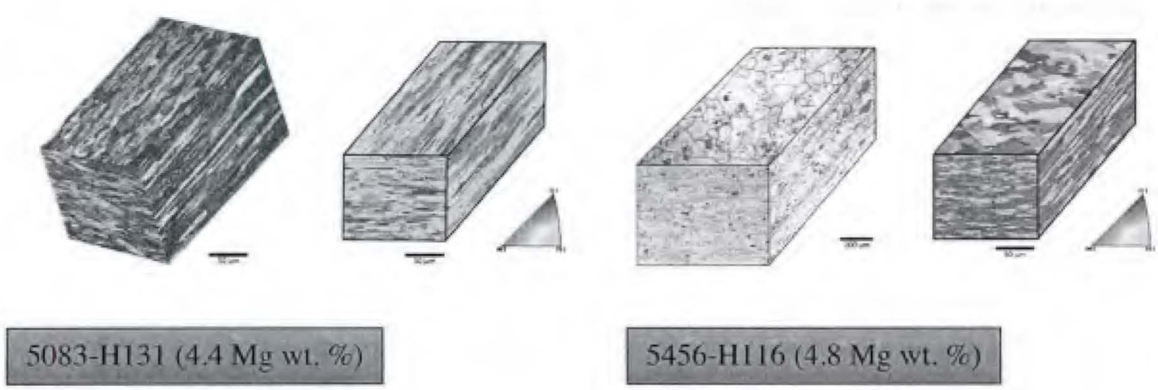


Figure 3. Optical 3D and EBSD images for as-received 5083-H131 and 5456-H116. Similar characterization was used for 5083-H116.

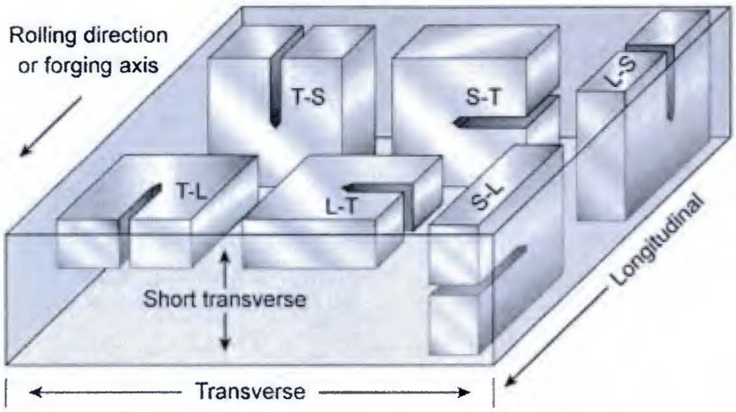


Figure 4. ASTM designations used to identify sample orientations tested in the different types of experiments in this grant.

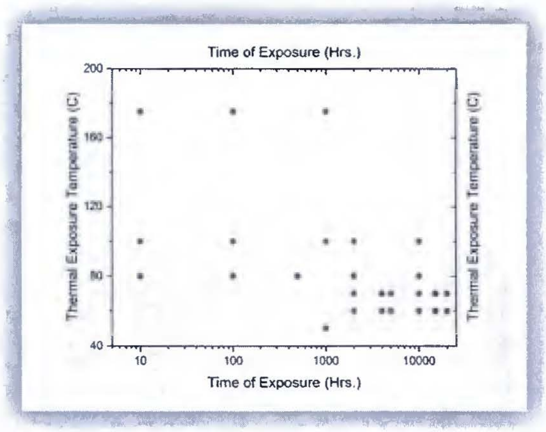
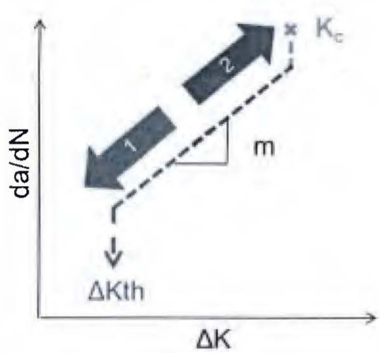


Figure 5. Thermal exposure conditions used to 'sensitize' samples. Both short term-high temperature and long term-low temperature exposures were conducted.



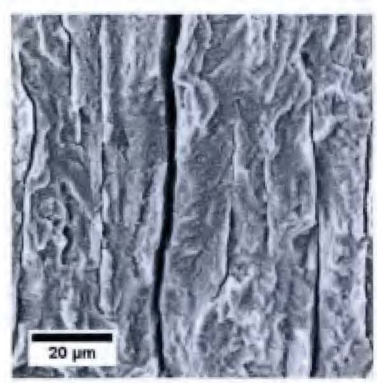


Figure 6. Fatigue crack growth experiments were conducted in accordance with ASTM standards (left image) on samples in the as-received and sensitized conditions for the L-T ASTM orientation. Sensitized samples exhibted grain boundary delamination perpendicular to the macroscopic crack plane (right image), indicative of degradation of properties in the short transverse (S) direction.

The effects of sensitization conditions, Figure 5, on fatigue crack growth behavior in humid air (e.g. RH 45%) and dry air (e.g. RH < 1%) was determined on L-T samples, Figure 4, taken from rolled plate in accordance with ASTM standards on both as-received and sensitized material in the manner shown in Figure 6 (left). Fracture surfaces were analyzed to determine the effects of sensitization on the fracture mechanisms at different regions of the fracture surface corresponding to the regions indicated in Figure 6 (left). The key observation was that sensitized materials often exhibited grain boundary delamination perpendicular to the crack growth direction, as indicated in Figure 6 (right). This indicates a severe degradation of properties in the short transverse (S) direction and required more work to directly probe fracture in the most susceptible orientation (e.g. S-T) by excising test samples (e.g. SSRT, FCG, and EAC tests) directly from the S-T orientation, Figure 4. The regimes of thermal exposure (i.e. time, temperature) and stress intensity, K, where such grain boundary delamination (i.e. splitting) was exhibited are summarized in Figure 7. It is clear that rapid sensitization and splitting is observed for the high temperature-short time exposures, while splitting is also exhibited for lower temperature exposures, but these require much longer exposure times (e.g. > 1,000 hours) to appear in the FCG humid air experiments.

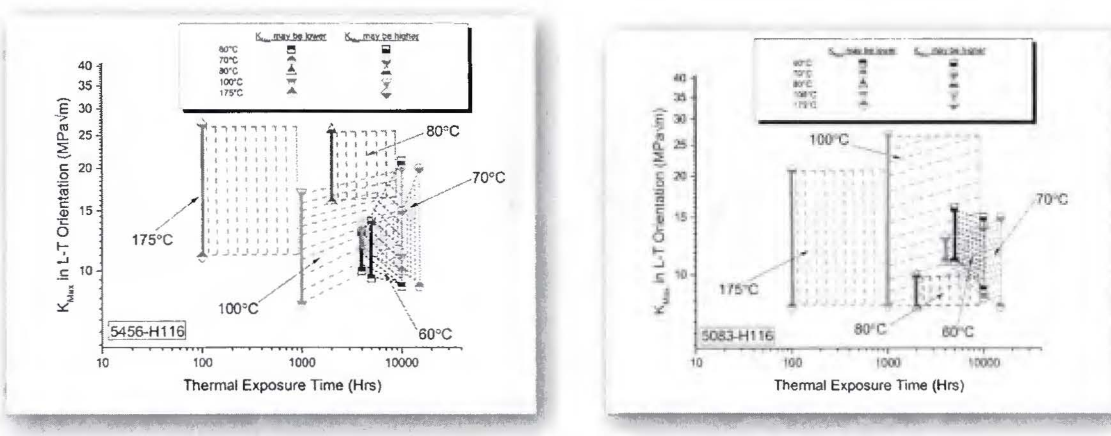


Figure 7. Regimes of grain boundary delamination (i.e. splitting) for 5456-H116 (left) and 5083-H116 (right) for samples tested in FCG in humid air after different thermal exposure conditions (i.e. time, temperature). The Kmax regime where splitting was exhibited is shown by the colored regimes. Similar maps have been generated for other 5XXX alloys, but less extensive in nature due to Navy primary interests in 5456/5083.

In addition to determining the K regimes of EAC in FCG shown in Figure 7, the locus of failure in the microstructure was also determined using the procedure shown in Figure 8. Figure 8 (left) shows the general appearance of the splits while Figure 8 (right) shows the approach to determine the locus of such grain boundary splits in the microstructure. The samples were first sectioned as shown in Figure 8 (right) to expose the splits in cross section. EBSD was then conducted in order to image the microstructure in the region of the splits as shown in Figure 8 (right). Analyses of mulitple EBSD results of this sort revealed that splits typically occurred between grain boundaries where there was a large gradient in Taylor factor. TEM and STEM also showed that Mg-segregration and Mg-rich phases were present on these boundaries.

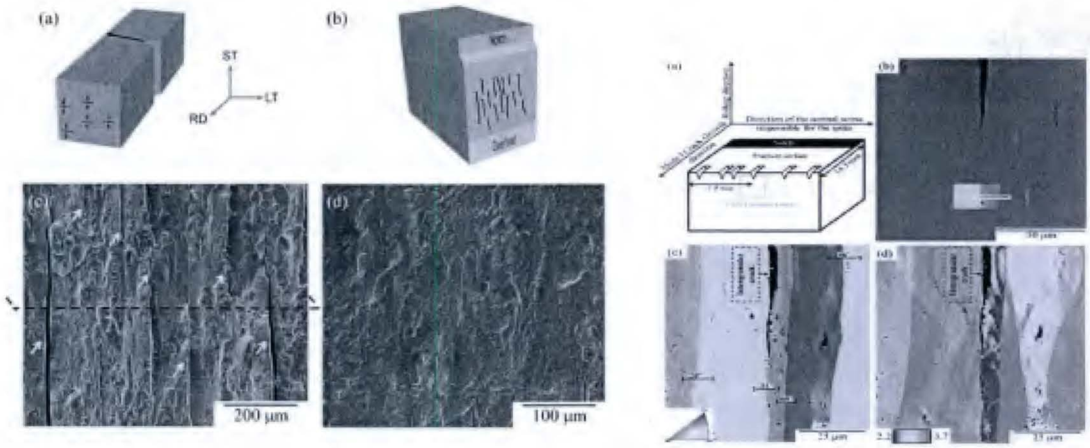


Figure 8. Schematic showing splits in senstized samples tested in FCG in humid air (left). Samples were sectioned in the manner shown at right, then EBSD was conducted to determine the locus of failure. TEM and STEM also revealed Mg-rich phases at the grain boundaries.

In addition to the extensive fatigue crack growth work summarized above and published elsewhere, extensive SSRT testing was conducted on samples taken from the S orientation as shown in Figure 9. The thermal exposure conditions are shown in Figure 9 as well as the stress vs. % elongation curves and that reveal severe EAC, summarized in Figure 9 (far right).

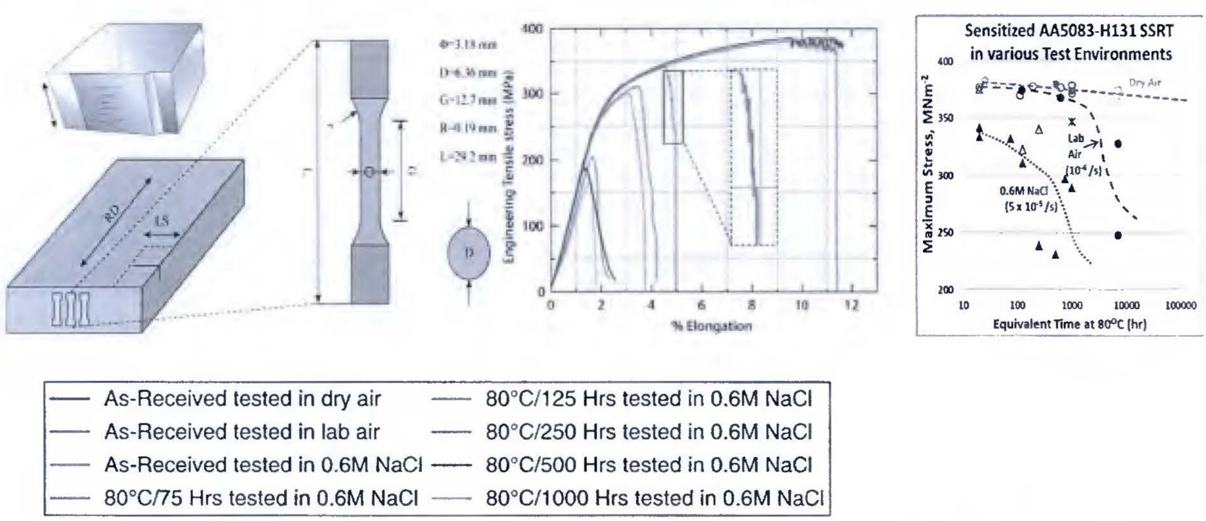


Figure 9. SSRT samples taken from S orientation and tested under the conditions shown. Stress vs. % elongation shown at right reveals severe effects of sensitization and test environment on EAC. The rightmost image quantifies the effects for many samples, exposure times, and environments.

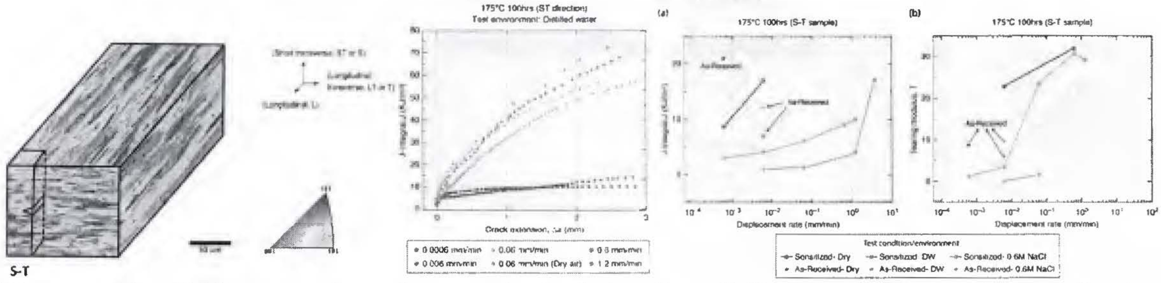


Figure 10. EAC experiments conducted in the S-T orientation. The effects of sensitization, loading rate, and solution, show the effects of these variables on both the initiation energy, Jlc and propagation energy, J, required to continue cracking.

The EAC resistance was also explored in the S-T plane by conducting fracture experiments on fatigue precracked samples in the manner shown in Figure 10 (left). Both the J vs crack length data (Figure 10, middle) as well as the effects of displacement rate on crack initiation and crack growth resistance are shown. Figure 10 (far right) shows the effects of exposure conditions and environment on both the energy for EAC initiation and EAC growth.

The work conducted under ONR N00014-14-1-0593 has produced a better understanding of the mechanisms of EAC in 5XXX alloys along with regimes of susceptibility. Follow on work is required to extend this work to longer term exposures as well as remediation approaches. These are being pursued.

References

Airlie 2004, Materials and Structures for Initiative in Advanced Ship Protection, August 30, September 2, 2004.

Rice, J. R., Continuum mechanics and thermodynamics of plasticity in relation to microscale deformation mechanisms. 1975.

Students Supported in this Work

Mohsen Seifi completed his PhD project with the funding provided by N000014-14-1-0593 and has continued some work as a Research Associate. In addition, Wenbin Gao, a visiting student from Tianjin University has conducted work on H128 sensitization-resistant material. Various undergraduate projects were also conducted. Adjunct Professor Henry Holroyd provided advice during the conduct of this work and there were various interactions with Prof. M. Taheri at Drexel and Dr. Golumbfskie at NSWC. The PI has visited all of these collaborators on various occasions.

Presentations Acknowledging N00014-14-1-0593

The following presentations acknowledged the funding provided by N00014-14-1-0593, starting with 2014 and ending with 2017. Eighteen (18) total presentations were made. Seven (7) of those presentations were invited and eleven (11) were contributed.

2014 (Designates Invited Talk)**

"Thermal Exposure Effects on Fracture and Fatigue of Al-Mg Naval Alloys", S.M. Seifi and J.J. Lewandowski, CWRU Research SHOWCASE, Cleveland, OH, April 18, 2014.

**"Microstructure and Mechanical Properties of Intermediate Temperature Sensitized Al-Mg Alloys", J.J. Lewandowski, Naval Surface Warfare Center, Bethesda, MD, July 25, 2014.

"Investigating the Temperature Dependence of Precipitation-Induced Mechanical Stability of Al-5456 and Al-5083 in Marine Environments", J. Gaies, W. Golumbfskie, J. Lewandowski, M. Taheri, UVa Corrosion Review, Charlottesville, VA, August 7, 2014.

"Sensitization Effects on Fracture and Fatigue of Al-Mg Naval Alloys", S.M. Seifi and J.J. Lewandowski, MS&T, Pittsburgh, PA, October 13, 2014.

2015

"Sensitization Effects on Fracture and Fatigue of Al-Mg Naval Alloys", S.M. Seifi, N.J.H. Holroyd and J.J. Lewandowski, TMS Annual Meeting, Orlando, FLA, March 18, 2015.

"Thermal Exposure Effects on Fracture and Fatigue of Al-Mg Naval Alloys", S.M. Seifi and J.J. Lewandowski, CWRU Research SHOWCASE, Cleveland, OH, April 17, 2015.

"Sensitization Effects on Fracture an Fatigue Behavior of Al-Mg Naval Alloys", M. Siefi, H. Jiang, B. Li, and J.J. Lewandowski, 3rd World Congress on ICME, Colorado Springs, CO, June 1, 2015.

**"Effects of Thermal Exposures on Fracture and Fatigue in 5XXX Alloys Tested in Different Orientations (and Environments)", J.J. Lewandowski, ONR Program Review, University of Virginia, Charlottesville, VA, August 14, 2015.

2016

"Effects of Test Environment and Sensitization on Fracture and Fatigue Behavior of Al 5XXX Series Alloys", M. Seifi, K. Macke, H. Holroyd, and J.J. Lewandowski, ASMI Cleveland Chapter Meeting, Cleveland, OH, January 25, 2016.

"Effects of Sensitization, Environment and Loading Rate on the Stress Corrosion Cracking Behavior of Al-Mg Alloys", S.M. Seifi, N.J.H. Holroyd, and J.J. Lewandowski, TMS Annual Mtg, Nashville, TN, February 16, 2016.

"Role of Sensitization on the Fatigue Crack Growth Behavior of Al-Mg Alloys Using ICME Approach", S.M. Seifi and J.J. Lewandowski, TMS Annual Mtg, Nashville, TN, February 16, 2016.

**"Environmental Effects on Fracture and Fatigue of Marine Alloys", J.J. Lewandowski, Mini-Symposium on Marine and Offshore Risk Engineering and Advanced Materials-CWRU, Cleveland, OH May 5, 2016.

**"Rate-Controlling Processes During Environment-sensitive Crack Propagation in Aluminum', T.L. Burnett, N.J.H. Holroyd, M. Seifi, and J.J. Lewandowski, International Workshop on the Environmental Damage in Structural Materials Under Static Load/Cyclic Loads at Ambient Temperatures", Engineering Conferences Int'l, Cork, Ireland, June 1, 2016.

**"Pre-exposure Embrittlement of Sensitized Al-Mg Alloy 5083-H116", N.J.H. Holroyd, T. Burnett, M. Seifi, and J.J. Lewandowski, International Workshop on the Environmental Damage in Structural Materials Under Static Load/Cyclic Loads at Ambient Temperatures", Engineering Conferences International, Cork, Ireland, June 2, 2016.

2017

"New Insights into Environment Assisted Cracking of Pre-Exposed and Sensitized 5xxx Series Aluminum", T. Burnett, H. Holroyd, J.J. Lewandowski, and M. Seifi, Corrosion 2017, New Orleans, LA, March 27, 2017.

"Pre-Exposure Embrittlement of Aluminum and Magnesium Alloys", J. Holroyd, T. Burnett, J.J. Lewandowski, and M. Seifi, Corrosion 2017, New Orleans, LA, March 27, 2017.

**"Thermal Exposure and Environment Effects on Tension, Fracture and Fatigue of 5XXX Alloys Tested in Different Orientations", J.J. Lewandowski, ONR Program Review, USC, Los Angeles, CA, May 24, 2017.

**"AM and Lightweight Alloys Research", J.J. Lewandowski, ARL Delegation Visit to CWRU, CWRU, Cleveland, OH, July 31, 2017.

Papers published acknowledging N00014-14-1-0593

Seifi, M., Samimi, P., Ghamarian, I., Collins' P.C., and Lewandowski' J.J. (2015). "Grain Orientation Effects on Grain Boundary Delamination During Fatigue of a Sensitized Al-Mg Alloy", Phil. Mag. Letters, 95(11), pp. 526-533.

Seifi, S.M., Holroyd, N.J.H., and Lewandowski, J.J. (2016). "Deformation Rate and Sensitization Effects on Environmentally Assisted Cracking of Al-Mg Naval Alloys", Corrosion, 72(2), pp. 264-283.

Holroyd, NJ.H., Burnett, T.L., Seifi, M., and Lewandowski, J.J. (2017). "Improved Understanding of Environment-Induced Cracking (EIC) of Sensitized 5XXX Series Aluminum Alloys", Materials Science and Engineering A, 682, pp. 613-621.

Holroyd, N.J.H., Burnett, T.L., Seifi, M., and Lewandowski, J.J. (2017). "Pre-exposure Embrittlement of a Commercial Al-Mg Alloy, AA5083-H131", Corrosion Reviews, 35(4/5), pp. 275-290.

Burnett, T.L., Holroyd, N.J.H., Lewandowski, J.J., Ogurreck, M., Rau, C., Kelly, R., Pickering, E.J., Daly, M., Sherry, A.H., Pawar, S., Slater, T.J.A., and Withers, P.J. (2017). "Degradation of Metallic Materials Studied by Correlative Tomography", in 38th Riso International Symposium on Materials Science – IOP Conf. Series: Materials Science and Engineering, 219, 021001.

Gao, W., Seifi, M., Wang, D., and Lewandowski, J.J. (2018). "Anisotropy of Corrosion and Environmental Cracking in AA5083-H128 Al-Mg Alloy", Materials Science and Engineering A, in preparation.

Seifi, S.M., Ghamarian, I., Samimi, P., Collins, P.C., Holroyd, N.J.H., and Lewandowski, J.J. (2018). "Sensitization and Remediation Effects on Evironmentally Assisted Cracking of Al-Mg Naval Alloys", Corrosion Science, in press.

Awards Received by PI and Students during 2014-2017 supported period

PI Awards: John J Lewandowski

Nominee - John S. Diekhoff Award for Graduate Mentoring, CWRU (2017)
Los Alamos National Lab Institute for Materials Science Invited Seminar Speaker (2015)
Gordon Conference on Physical Metallurgy Speaker (2015)
Summer Visiting Professor-Singapore Center 3D Printing (SC3DP), Singapore (2015-present)
TMS Leadership Award (2014)

G Student Awards: Mohsen Seifi, Wenbin Gao

Mohsen Seifi

- 2014 ASTM Graduate Scholarship
- 2014 ASM International Presidents Award
- 2014 TMS DeWitt-Smith Scholarship
- 2015 ASTM Graduate Scholarship

Wenbin Gao

- China Student Scholarship Award

Visiting Scholar: Wenbin Gao (Tianjin University)